Phase fraction evolution in a multi-phase intermetallic γ-TiAl based alloy

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The continuous demand for weight reduction and higher engine efficiencies in automotive, aerospace and energy industries pushes the currently applied materials towards their limits. Therefore, these industries have a strong need for the development of novel lightweight materials, which can withstand temperatures up to 800 °C, while maintaining acceptable mechanical properties. Intermetallic γ-TiAl based alloys, such as TNM™ alloys, are among the most promising candidates, which possess these required thermal and mechanical properties [1].

For the current investigations TNM material with a composition Ti-43.9Al-4.0Nb-0.95Mo-0.1B (at%) was produced by argon gas atomization [2] and hot-isostatic pressing (HIPing) at 1250 °C for 2 h at 200 MPa followed by furnace cooling. Starting from the as HIPed condition heat treatments were performed in a Carbolite furnace RHF 1600 by subsequent oil quenching (OQ). The heat treated samples were ground and etched electrolytically [3] in order to evaluate the phase fractions by XRD measurements in a Bruker-AXS D8 Advance Diffractometer in Bragg-Brentano geometry using Cu-K_α radiation. Rietveld analysis was performed with the commercial software package TOPAS by Bruker-AXS, Madison, USA. The microstructural constituents were analysed with a light-optical microscope (LOM) from the heat treated samples. Thereby, the samples were additionally colour-etched electrolytically using an Ence and Margolin solution [4] and were examined with a LOM from Zeiss, Type Axio Imager M1m. The evaluation of phase fractions with LOM was performed using the commercial software AnalySIS from Olympus Soft Imaging System GmbH, Germany. For the in-situ synchrotron experiments the high-energy X-ray diffraction (HEXRD) setups of the HZG beamline HARWI II at DESY in Hamburg, Germany, were used [5-7]. Specimens with a diameter of 5 mm and a length of 15 mm were heated in a modified quenching and deformation dilatometer Bähr DIL 805 A/D from Bähr-Thermoanalyse GmbH under Ar-atmosphere [8]. For the 0.5 x 0.5 mm^2 beam a mean energy of 104.7 keV was adjusted. The samples were heated to 1000 °C, held for 10 min, and then continuously heated at a rate of 2 K/min to 1350 °C. A mar555 detector by Marresearch GmbH, Norderstedt, Germany, was employed. The azimuthal integration of the acquired diffraction patterns was performed with the software fit2D [9]. For the prediction of the prevailing phases, their fractions and transition temperatures thermodynamic equilibrium calculations based on CALPHAD (calculation of phase diagrams) were conducted with the software MatCalc© using a commercially available database for TiAl [10]. More detailed information regarding the whole investigation is given in [11].

The calculated phase fraction diagram is shown in Fig. 1a. In recent publications, however, the thermodynamic database used here was found to describe the transition temperatures and phase proportions poorly as reported in [12,13]. Therefore, the calculation shown in Fig. 1a expresses trends for phase fractions and transition temperatures rather than absolute values. The experimentally evaluated phase fractions from LOM images, ex-situ XRD and in-situ HEXRD are summarized in Fig. 1b. From Fig. 1b it is evident that the phase fractions obtained from LOM images of the heat treated samples are in agreement with the in-situ HEXRD results up to $T_{\gamma solv}$, which refers to the dissolution temperature of the γ-phase. At temperatures above $T_{\gamma solv}$, however, the HEXRD results show a higher β-phase fraction than detected by LOM. Obviously, it was not possible to freeze-in the whole β-phase fraction. It is supposed that the high cooling rate provided by OQ triggers a martensitic β→α transformation. An increase in holding time from 1 h to 10 h at temperatures higher than 1200 °C shows only a decrease of about 2 vol.% in the experimentally observed phase fraction of the β-phase, determined by XRD as well as LOM. This observation, in combination with the HEXRD results at a heating rate of 2 K/min, leads to the conclusion that dissolution and precipitation kinetics of the β-phase in this temperature region are fast. Only a small difference in the γ-solvus temperature can be recognized between the calculated and experimentally

derived data. The course of the phase fractions around $T_{\gamma solv}$ is in agreement with the calculated data. The calculated and measured phase fractions above and below $T_{\gamma solv}$ differ increasingly with increasing distance to $T_{\gamma solv}$. The existence of the predicted small single α-phase field was experimentally confirmed by the results of the 1 h heat treatments between 1260 °C and 1265 °C as well as in in-situ HEXRD investigations. Altogether, these results show that thermodynamic calculations reflect the qualitative trends of the temperature dependence of phase fractions around $T_{\gamma solv}$ rather well. Nevertheless, for more exact quantitative calculations the thermodynamic database for TiAl with high Nb (and Mo) contents must be further improved, as recently reported for Nb-rich γ-TiAl alloys [13,14].

It can be concluded that in-situ HEXRD experiments are a fast and reliable method to evaluate phase fractions in complex multi-phase materials in order to obtain quantitative information about the phases prevailing at different temperatures and to verify thermodynamically calculated data.

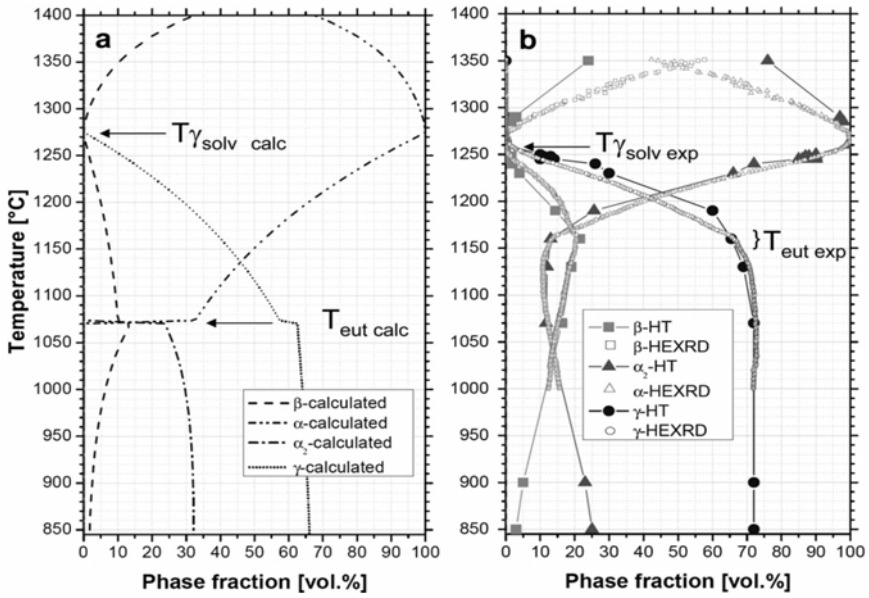

Figure 1: a) Calculated phase fractions for Ti-43.9Al-4.0Nb-0.95Mo-0.1B. b) Results of quantitative analysis of heat treated samples (solid symbols), compared with in-situ HEXRD measurements (open symbols) [11].

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